Semisolid Microstructural Evolution of AlSi7Mg Alloy During Partial Remelting

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Abstract

Different as-cast microstructures of an AlSi7Mg alloy were produced by controlling the solidification conditions. The as-cast grain size ranged from 1.4 mm to 160 μm and the morphology varied from dendritic to rosette-like to globular. The as-cast materials were then partially remelted and isothermally held at 580°C for microstructure evolution. The final microstructure depended on the initial as-cast microstructure and the isothermal holding time. After partial remelting and isothermal holding, coarse-grained dendritic structures were not able to evolve to a globular structure, while structures with medium sized dendritic grains evolved to a globular structure with a relatively large particle size after a long isothermal holding time. Fine-grained structures evolved to well-rounded globular grains within times ranging from 10 minutes to 5 minutes as the dendritic nature of the starting structure diminished. An empirical equation has been established to describe the relationship between the evolved microstructure and the as-cast microstructure.

Keywords: Semisolid processing; Microstructural evolution; Thixotropic feedstock; AlSi7Mg alloy

1. Introduction

Semisolid metal processing is an attractive technology for metal forming. It uses neither fully solid metal such as in forging and extrusion, nor fully liquid metal such as in casting. Instead, it uses semisolid slurries, which are mixtures of globular solid particles uniformly suspended in a liquid matrix. Semisolid slurries with such a microstructure exhibit thixotropic and pseudoplastic behaviour, allowing them to be handled as a solid when at rest and having fluid-like properties when sheared during the forming operation. Semisolid metal processing offers to industry the ability to cast components of complex shape, substantially higher in quality than die castings but lower in cost than forgings [1-5]. The process is feasible only when the starting material has a non-dendritic microstructure [4-6], and several methods have developed to produce feedstock materials with such a microstructure [1,7-11]. Among them mechanical stirring and electromagnetic stirring have been proven as efficient means of producing non-dendritic structures, as stirring during solidification inhibits the formation of a dendritic structure [1,12,13]. However, special stirring equipment is needed in these methods.

One alternative is semisolid thermal transformation, which relies on the ripening of a more or less dendritic structure when it is heated and held for a period of time in the semisolid temperature range [14-19]. The microstructural evolution depends on the starting structure of the materials and on the semisolid remelting process. The microstructural evolution that occurs during partial remelting of feedstock prior to casting may be an incidental consequence of reheating an already adequate microstructure, or it may be a necessary part of the process, without which the microstructure is not suitable for semisolid forming. In this work, a variety of as-cast microstructures with different grain sizes and morphologies have been produced. The microstructures were partially remelted and isothermally held in the semisolid condition. The effect of the as-cast microstructure and the partial remelting process on the microstructural evolution is investigated, and a relationship between the as-cast microstructure and the evolved microstructure is established.

2. Experimental procedure

The base alloy used in this study was a commercial AlSi7Mg0.35 alloy. The ingot was charged in an induction furnace. The melt was raised to 750°C, then degassed for 10 minutes with argon bubbled through a graphite lance. The melt was then allowed to cool to the chosen pouring temperature, and cast into a steel mould that had been preheated to 150°C.

Small cylindrical samples (20 mm in diameter and 10 mm height) were cut from the castings. The samples were first heated in an electric resistance furnace preset at 650°C, giving a heating rate of 1-2°C/s. Once the samples reached 550°C, they were quickly transferred to a molten salt-bath set at 580°C. The use of the preheat furnace minimised the thermal load on the salt bath. A K-type thermocouple was inserted into the center of the samples to monitor the temperature during heating. A salt bath was used for rapid heating and good thermal contact. The heating rate was about 15°C/s. The isothermal holding times were measured starting from the time the sample temperature first reached 3°C from the bath set point. Samples were isothermally held in the salt bath for 3, 8, 15 and 30 minutes, then quenched in water for microstructural examination.

As-cast samples and partially remelted samples were sectioned and polished. They were anodized in a solution of 760 mL water and 40 mL fluoroboric acid at 20 volts for about 30 seconds. Grain size was measured using the linear intercept method outlined in section E112-88 of ASTM standards, 1994. Some remelted samples were etched in 10% H_3PO_4 solution at 50°C for 3 minutes to highlight the contrast between the eutectic phase and the primary phase. The microstructures were characterized by measuring the particle size and shape of the primary α-phase on two-dimensional sections. An automatic image analysis system (Cambridge Instruments Quantimet 570) was used to measure each α-phase particle cross-

sectional area A_j and perimeter P_j. The particle size was characterized by its equivalent circle diameter, defined as: $2(A_j/\pi)^{1/2}$, and the particle shape was characterized by roundness, defined as: $P_j^2/(4\pi A_j)$. Roundness varies from 1, for a microstructure having perfectly spherical particles to very large numbers, such as 50, for highly branched or elongated microstructures. In order to obtain a statistically valid result, at least 500 particles were counted in each specimen. For these measurements it was assumed that the boundary between solid and liquid in the semisolid state was defined by the boundary between α-phase grains and areas of eutectic in the quenched microstructures.

3. Results

3. 1. As-cast microstructure

Fig. 1 shows the initial as-cast microstructures of castings produced by the controlled-pouring method. The grain size and morphology varied significantly with the pouring conditions. Fig. 1(a) shows the microstructure from quench-casting in which a thin-walled steel tube was used as the mould and was quenched into water after melt pouring. The rapid cooling condition resulted in a fine dendrite arm spacing, but the lack of mould chill resulted in large grains – about 1400 μm. The microstructure is described as "dendritic and very coarse-grained". Most of the eutectic phase was intragranular, distributed between the dendrite arms. Figs. 1(b) to 1(e) show the microstructures from permanent mould castings. Compared to quench-casting, the grain size was much smaller and the grain morphology was less branched, although dendrite arm spacings were similar.

Fig. 1(b) shows the microstructure of the sample from high-temperature-pouring. It had a coarse-grained dendritic structure with grain size 900 μm and dendritic morphology. Fig. 1(c) shows a medium-grained dendritic microstructure from a casting using a medium pouring temperature. The grain size was approximately 350 μm. Low-temperature-pouring resulted in fine grains, as shown in Figs. 1(d) to 1(f). The grain size was about 160-200 μm. The grain morphology varied with the different solidification conditions from a dendritic structure (Fig. 1(d)), to a rosette-like structure (Fig. 1(e)), to a globular structure (Fig. 1(f)). Most of the eutectic phase was intergranular. Table 1 summarises the microstructural characteristics observed. The materials made by low-temperature-pouring method have excellent microstructural homogeneity. In contrast, the electromagnetically stirred material has been reported to be nonuniform from the billet surface to centre and the primary α-phase may have a bi-modal appearance where fine rosette structure coexist with coarse grains up to 300 μm in size [3, 13].

The material with dendritic morphology, although found in a large range of grain sizes, was not observed to vary in secondary dendrite arm spacing within the range of casting conditions used here. For this reason, throughout this paper references to coarse, medium and fine structures will refer to grain size. Medium-sized and larger grains always exhibited dendritic shapes. Only the material with finer grain sizes needs to be discriminated by morphology.

3.2. Microstructural evolution

The as-cast materials were partially remelted and isothermally held at 580°C. For AlSi7Mg alloy, at this temperature the eutectic phase is remelted while almost all the primary phase remains solid. The materials were isothermally held at 580°C for 3, 8, 15 and 30 minutes. During the isothermal holding stage the microstructure evolves, driven by the reduction of interfacial area between the solid and liquid phases.

Fig. 2 shows the microstructural evolution of the very coarse-grained material obtained by quench-casting. Liquid remelting occurred along both grain boundaries and dendrite arms. After 3 minutes holding, coalescence of the dendrite arms had started to occur and the

dendritic appearance started to diminish. However, the grain size remained unchanged with almost all of the liquid being intragranular (Fig. 2(a)). After 8 minutes of isothermal holding, the coalescence of the dendrite arm resulted in the disappearance of the dendritic structure. In the two-dimensional section, each grain evolved to a solid particle surrounded by a relatively uniform thin liquid film, and within the particle a large number of small liquid regions about 10-30 μm in diameter were dispersed (Fig. 2(b)). These liquid pockets appeared isolated in the 2D section but were possibly interconnected out of the plane of section. With increasing holding time, the solid phase ripened. As a consequence, small liquid pockets merged and some of them linked to form a liquid network, but it remained substantially intragranular. The liquid region in grain boundaries also became thicker. The solid phase evolved to a rounded morphology, forming an interconnected solid network within each grain (Fig. 2(c)). After prolonged isothermal holding for 30 minutes (Fig. 2(d)) the solid phase became more rounded but remained far from globular. There appeared to have been some breakup of solid within grains – i.e. the length of continuous liquid film increased. This initially seems that it would lead to an energetically unfavourable increase in surface area, however there will be a concomitant decrease in the volume and surface area of internally trapped liquid, which will account for this apparent anomaly.

Fig. 3 shows the microstructural evolution of the fine-grained dendritic structure produced by low-temperature-pouring. After 3 minutes of holding at 580°C (Fig. 3(a)), the structure had lost much of the initial dendritic morphology, and some grains had become rosette-like. After 8 minutes, the material became globular in structure. A high proportion of the liquid was intergranular, however, there was still some liquid enclosed in the solid particles in the form of many small liquid pockets, Fig. 3(b). After 15 minutes isothermal holding, the microstructure became quite globular and uniform in particle size, with an average diameter of about 100 μm (Fig. 3(c)). Further holding resulted only in structural coarsening with a slight increase in average particle size, Fig. 3(d).

The evolution of grain/particle size and roundness as a function of isothermal holding time is shown in Fig. 4 for the fine-grained dendritic structure. The as-cast material had a grain size of 200 μm. The microstructural evolution first proceeded with a slight increase in the grain size, followed by a decrease. The particle size first reduced slowly to 180 μm, then decreased rapidly to 100 μm, and the particles became globular. Finally with prolonged isothermal holding, the α-phase particle size started to increase very slowly. The significant apparent decrease in particle size was to some extent because the evolved solid particles were more compact than the initial dendrites. In the as-cast microstructure, the grain structure was dendritic with eutectic distributed between dendrite arms. The measured grain size, using the linear intercept method, regarded all the eutectic as being within grains. On the other hand, for the remelted microstructure, most of the eutectic became intergranular liquid. Particle size measured by image analysis excluded all the intergranular eutectic. Dendrite fragmentation during microstructural evolution probably also accounts for some of the decrease in the grain size.

The change of particle roundness with isothermal holding time occurred much more abruptly than the change of particle size. The starting material had a dendritic microstructure with a high roundness value of 15.7. The particle morphology changed rapidly at the beginning of isothermal holding. The microstructure lost its dendritic features and changed to rosette-like in the first 3 minutes of holding. The particle roundness changed to 3.4. As isothermal holding continued, the microstructure became more globular. Based on microstructural examination, it was decided that a roundness of 1.8 defined a sufficiently globular structure. For lower values, there was no apparent distinction to the eye. Therefore the material was estimated to take 10 minutes to evolve to a globular structure. After this, the particle roundness changed slowly with holding time. After 30 minutes holding, the roundness was 1.3.

3.3. Evolved Microstructures

Fig. 5 shows the evolved microstructures from the different as-cast microstructures after isothermally holding at 580°C for 15 minutes. The very coarse structure from quench casting formed a solid network and nearly all the liquid phase was intragranular (Fig. 5(a)). The coarse structure from high-temperature-pouring formed very irregularly shaped solid particles, some of which might be interconnected in three dimensions. About 25% of the eutectic was intragranular, Fig. 5(b). The medium-sized grains developed into a globular structure during isothermal holding, as shown in Fig. 5(c). The interconnected nature of the solid phase disappeared, with solid α-phase particles being rounder and separated from each other by eutectic. A large proportion of eutectic was intergranular with some entrapped in the solid particles. The fine structures from low-temperature-pouring all developed into a similar, clearly globular, structure, Figs. 5(d) to 5(f). Solid globules with an average size of about 100 μm were uniformly distributed in the eutectic matrix. Nearly all of the eutectic was intergranular. The structure appeared similar to that of material made by electromagnetic stirring [3].

4. Discussion

4.1. Relationship between the as-cast microstructure and the evolved microstructure

Microstructural parameters of the as-cast materials and their evolved structures after isothermal holding are summarised in Table 1.

The as-cast microstructure was varied by controlling the casting method and solidification conditions, and could be classified into three categories. These are illustrated in Fig. 6 which shows the effect of partial remelting at 580°C for 15 minutes on the particle size. Coarse and very coarse microstructures, which were obtained by quench casting and conventional permanent mould casting (high-temperature-pouring), had an as-cast grain size larger than 800 μm. After partial remelting and isothermal holding, the dendrite arms became rounded and coarsened. The evolved microstructure formed a solid network, far from globular, even after 15 minutes holding. Almost all of the liquid was intragranular, distributed throughout the solid network. This agrees with the results from Loue & Suery [15] who isothermally held a direct chill (DC) cast sample at 580°C for up to 27 hours.

A medium structure with a grain size between 200 and 600 μm was produced at a medium pouring temperature. After partial remelting and isothermal holding, such an equiaxed structure initially evolved to an irregular globular structure. After a long holding time it eventually evolved to a spherical globular structure, however the final particle size was relatively large. A coarse globular structure results in poor rheology, and excessive transformation times would be impractical and uneconomical for actual industry production.

Fine structures with a grain size less than 200 μm can be produced by low-temperature-pouring. Depending on the solidification conditions, the grain morphology of the fine structure could be dendritic, rosette-like or globular. Fine structures evolved to a spherical globular structure with a particle size of around 100 μm within an acceptable isothermal holding time. The microstructure with the more globular nature took less time to evolve to a spherical globular structure.

For those structures that could evolve to a globular shape (grain size less than 600 μm), the time for globularisation depended on the initial grain size and grain morphology in the as-cast microstructure. Since grain morphology and diameter were not independent variables under the experimental conditions in this study, grain size was chosen as the independent parameter for Fig. 7. This Fig. presents the globularisation time (the time required for particle roundness to reach 1.8) for the different structures produced in this study. The globularisation time decreased from 30 minutes to 10 minutes when the as-cast grain size changed from 350 μm to 200 μm. For the structures with a grain sizes less than 200 μm, the globularisation time

changed from 10 minutes for a fine dendritic structure to 8 minutes for a fine rosette-like structure and 5 minutes for a fine globular structure. Fig. 7 indicates that the globularisation time decreased almost linearly with the grain size. If the line is extended, it meets the X-axis at about 110 μm. This suggests that, if the microstructure is as fine as 110 μm, the material will have a suitable globular structure as soon as it is partially remelted. This value corresponds well to the grain size of the current commercial semisolid feedstock products.

4.2. Prediction of the evolved microstructure from the as-cast microstructure

The evolved microstructure was determined by the as-cast microstructure and the microstructural evolution process. Particle roundness was the most important parameter in evaluation of the suitability of a structure for semisolid forming. Fig. 4 indicated that particle roundness decreased monotonically with an increase of isothermal holding time. Based on the experimental results given in Table 1, a particle roundness equation could be established by including the as-cast grain size and the isothermal holding time, as described as follows:

$$Remelted\ \ Particle\ Roundness = 1 + \alpha \cdot \frac{(grain\ size_{as-cast} - grain\ size\ offset)^{\beta}}{(holding\ time + holding\ time\ offset)^{\gamma}} \qquad (1)$$

Grain size offset was defined from Fig. 7 as 110 μm, where it was shown that an as-cast grain size of 110 μm needed no extra globularisation time. A holding time offset was added because a small extent of microstructure evolution occurred during the reheating of the as-cast material. Holding time offset was set to be 0.5 minutes. So Eq. (1) transformed to:

$$Remelted\ \ Particle\ Roundness = 1 + \alpha \cdot \frac{(grain\ size_{as-cast} - 110)^{\beta}}{(holding\ time + 0.5)^{\gamma}} \qquad (2)$$

Grain size is in μm and holding time is in minutes. Using multiple linear regression after converting to a logarithmic form to fit the data, α was found to be 0.09, while β and γ were both close to 1, within estimated uncertainties. It was found that the values of α, β and γ were not very sensitive to holding time offset and grain size offset. Eq. (2) could be written as:

$$Remelted\ \ Particle\ Roundness = 1 + 0.09 \cdot \frac{grain\ size_{as-cast} - 110}{holding\ time + 0.5} \qquad (3)$$

Fig. 8 shows the particle roundnesses of evolved microstructures calculated for a range of as-cast microstructures using Eq. (3). The degree of microstructure globularisation was predicted based on the grain size of the as-cast microstructure (the grain size was linked to the grain morphology) and the isothermal holding time. Smaller as-cast grain size and longer isothermal holding time resulted in a smaller value of the particle roundness in the evolved microstructure (eg. a more globular evolved microstructure).

One application of the equation is to predict the evolved microstructure from the as-cast microstructure. For example, for an as-cast microstructure with a grain size of 250 μm, after 15 minutes of isothermal holding, the particle roundness will be 1.9. Another application of the equation is to calculate the globularisation time for a given as-cast microstructure. For example, for structures with grain sizes of 160 μm, 180 μm and 200 μm, the globularisation times are calculated as 5.3, 7.7 and 10.3 minutes respectively.

Although the predictive equation is built upon the experimental data and has not been independently verified, the predictions fit well across the entire range of data. This consistency, together with the relative simplicity of the equation, suggests that the relationship might be more broadly applicable. However, we emphasise that these results were determined for a single alloy system under a certain set of experimental conditions.

Further work would be required to ascertain whether (a) the general form of the equation applies to other alloys and (b) if so, what the parameter values are.

5. Conclusions

(1) Microstructural evolution occurs during partial remelting and isothermal holding. Dendritic grains with a diameter larger than 800 μm evolve to a rounded but irregular morphology and they are not able to evolve to a globular structure.

(2) Dendritic grains with a size between 200 and 600 μm evolve to a globular structure after a long time of isothermal holding and the resultant particle size is relatively large.

(3) A fine microstructure with a grain size less than 200 μm evolves to a spherical globular structure after a short isothermal holding time. The time taken for the material to be globular depends on the initial grain morphology and grain size. It changes from 10 minutes for a fine dendritic structure to 8 minutes for a fine rosette-like structure and 5 minutes for a fine globular structure.

(4) It was shown that an as-cast grain size of 110 μm or less requires no isothermal holding time and is suitable for semisolid casting once the casting temperature is reached.

(5) The evolved microstructure depends on the initial as-cast microstructure and the evolution process during isothermal holding. Smaller as-cast grain size and longer isothermal holding time result in a more globular microstructure. For the alloy and the experimental condition studied, the relationship between the as-cast and remelted microstructures can be described as:

$$Remelted\ Particle\ Roundness = 1 + 0.09 \cdot \frac{grain\ size_{as-cast} - 110}{holding\ time + 0.5}$$

This equation can be used to determine the isothermal holding time needed to achieve a suitable semisolid state as measured by particle roundness, for a particular as-cast feedstock grain size. Alternatively, it can be used to predict the state of the semisolid microstructure for any starting as-cast grain size after a fixed isothermal holding time.

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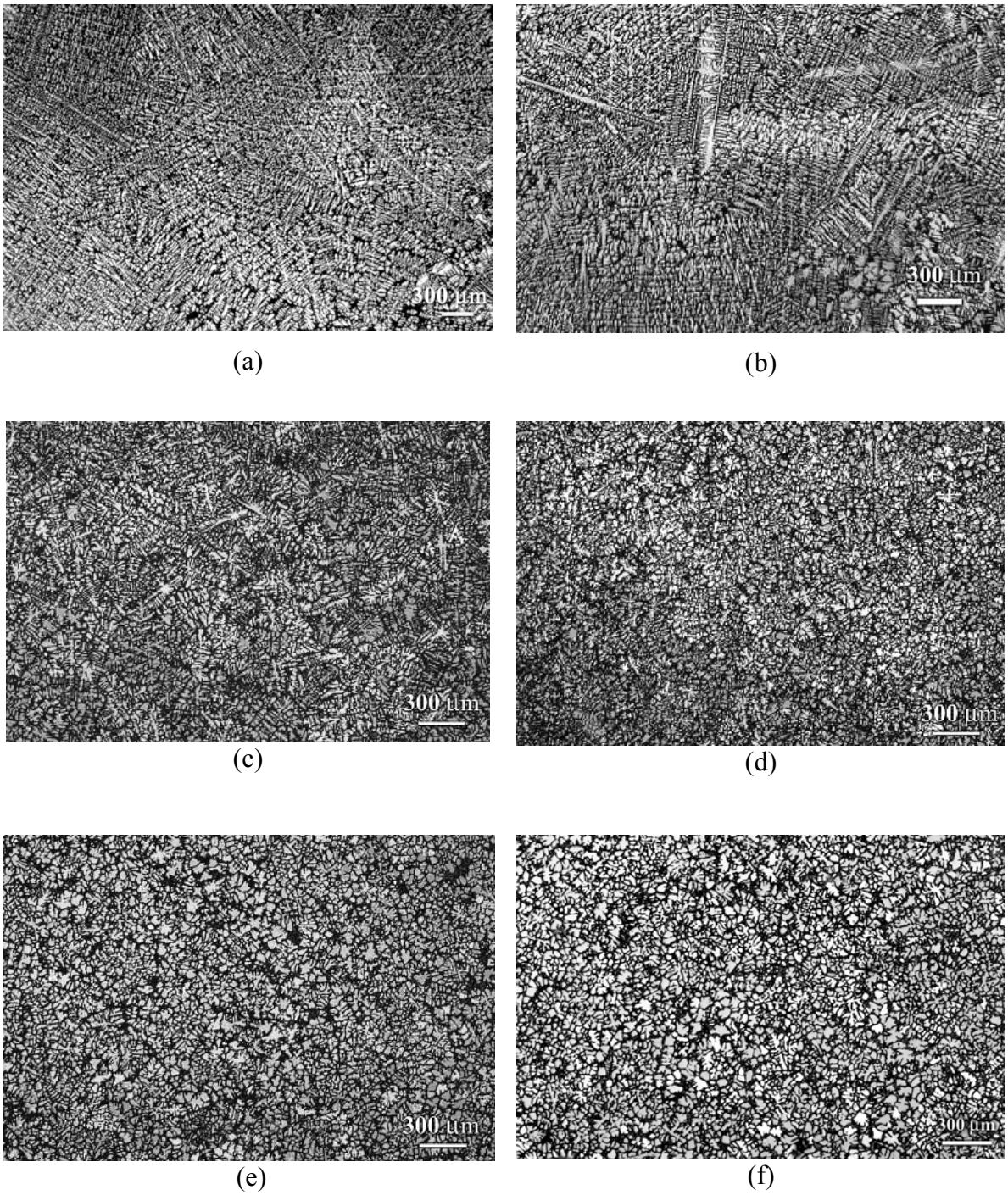


Fig. 1. As-cast microstructures: (a) very large grain structure produced by quench casting; (b) coarse structure from high-temperature-pouring (725°C); (c) medium structure from medium-temperature-pouring (675°C), and fine-grained structures of dendritic (d), rosette-like (e) and globular (f) morphologies from low-temperature-pouring (650°C or lower).

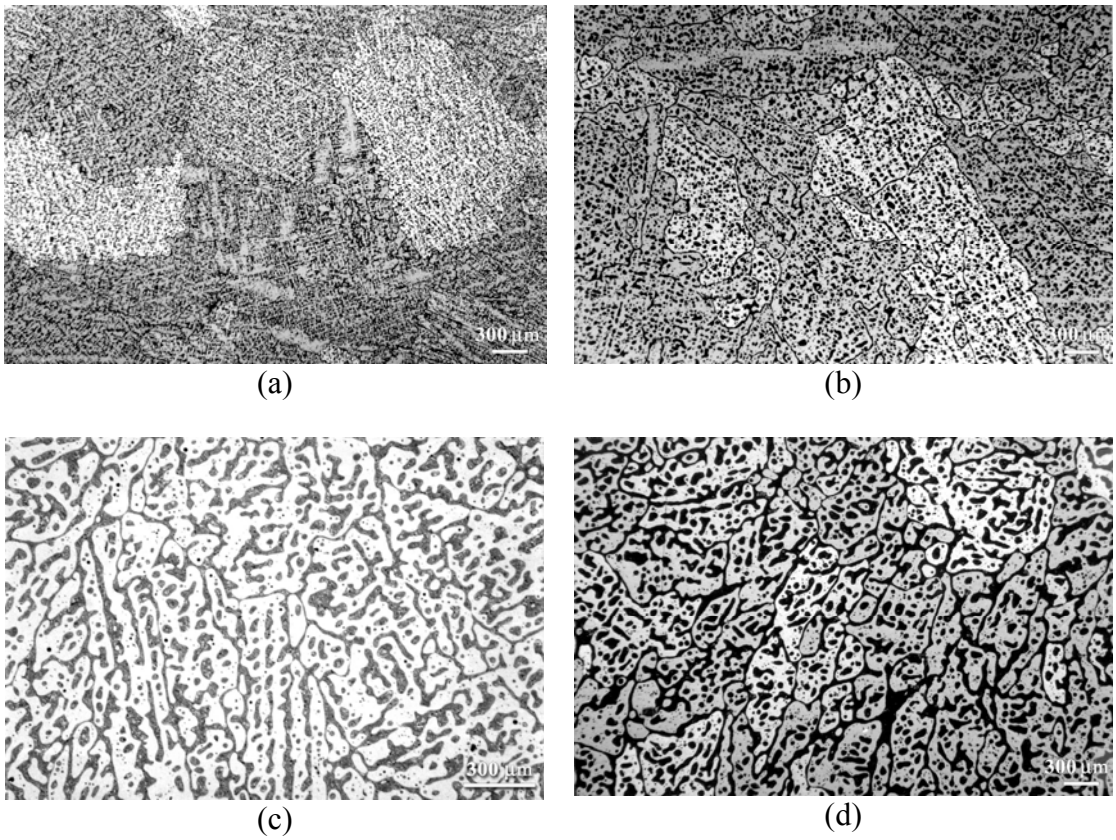
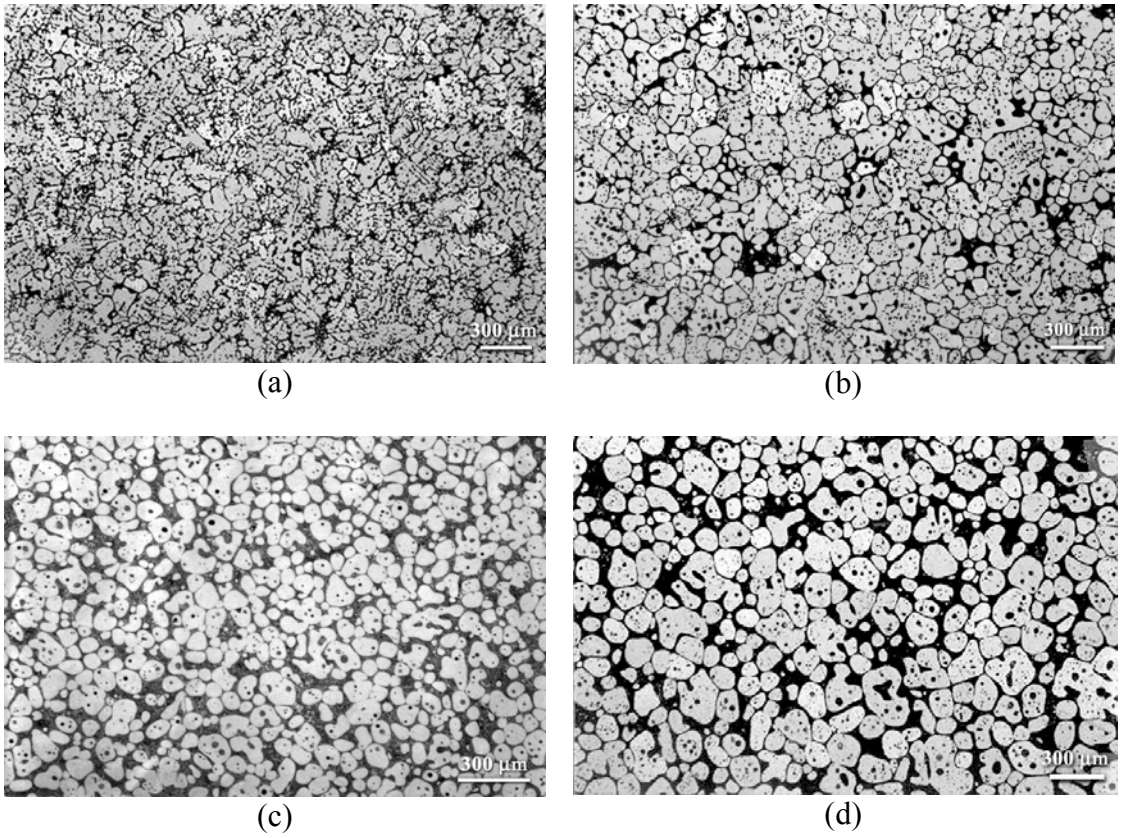


Fig. 2. Microstructural evolution of the very coarse-grained structure during partial remelting. Samples were isothermally held at 580°C for (a) 3 minutes; (b) 8 minutes; (c) 15 minutes and (d) 30 minutes.



(a) (b)

(c) (d)

Fig. 3. Microstructural evolution of the fine-grained dendritic structure during partial remelting. Samples were isothermally held at 580°C for (a) 3 minutes; (b) 8 minutes; (c) 15 minutes and (d) 30 minutes.

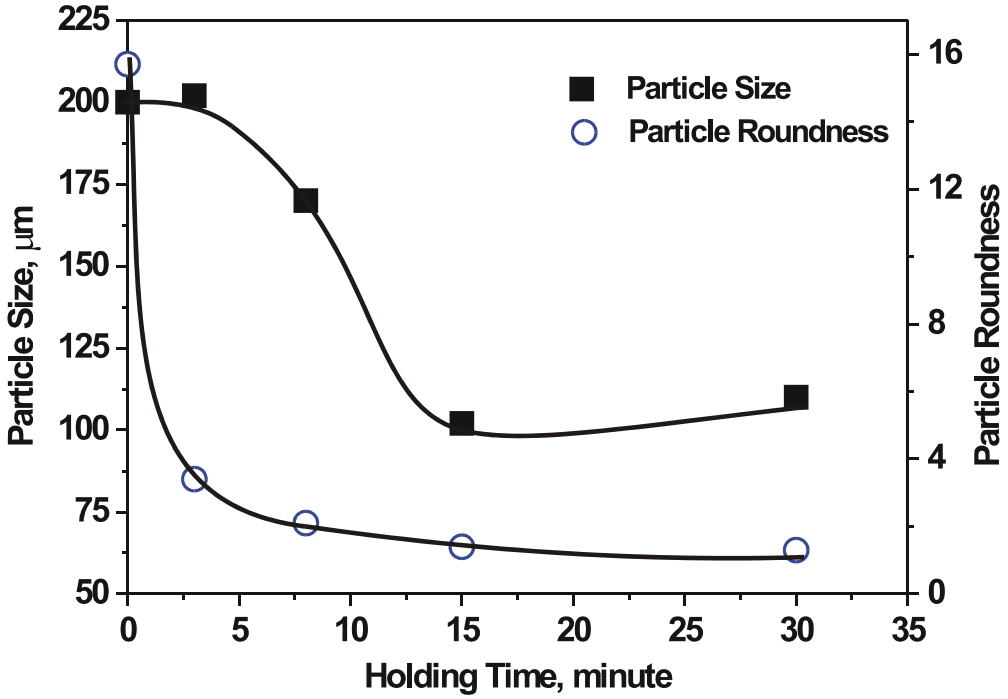


Fig. 4. Evolution of grain (or particle) size and roundness of a fine-grained dendritic structure as a function of isothermal holding time.

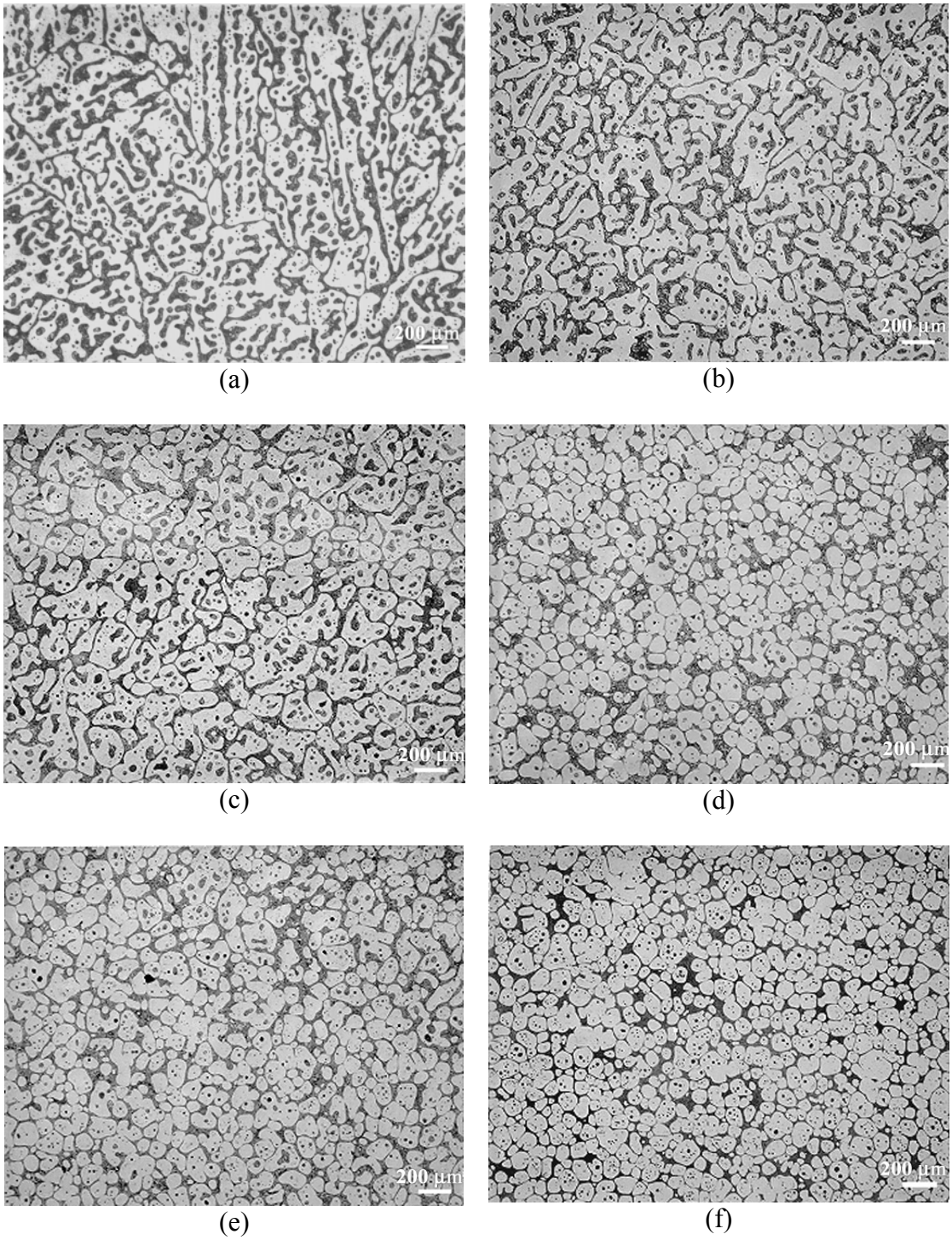


Fig. 5. Evolved microstructures produced after partial remelting and isothermal holding at 580°C for 15 minutes. The initial microstructures are (a) very coarse-grained structure; (b) coarse structure; (c) medium grain; (d) fine dendritic; (e) fine rosette-like and (f) fine globular.

Fig. 6. The grain size of the as-cast microstructure plotted against the particle size of the evolved microstructure. Microstructure evolution was carried out by isothermal holding at 580°C for 15 minutes. (■) very coarse-grained structure produced by quench casting, (♦) coarse structure from Loue & Suery [15], (●) coarse structure produced by 725°C pouring, (▲)medium-grained structure produced by medium temperature pouring and open symbols are structures produced with low temperature pouring.

Fig. 7. Time to globularisation (roundness <1.8) for different microstructures cast under the conditions as indicated. The isothermal holding was at 580°C.

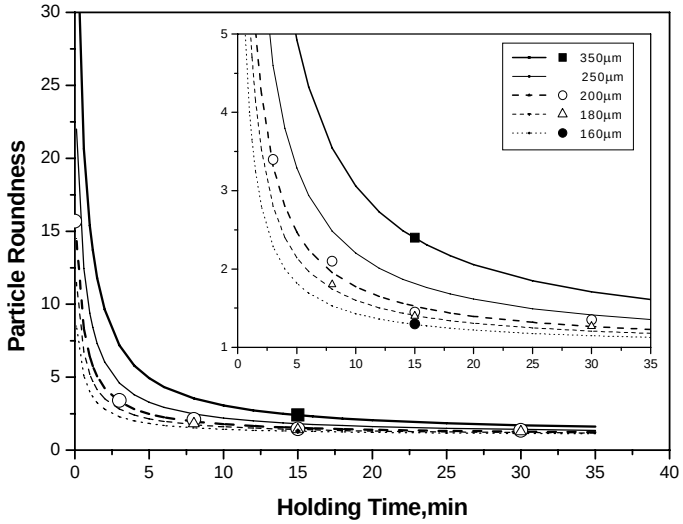


Fig. 8. Prediction of the particle roundness of remelted microstructures for a range of as-cast structures. The experimental results are also presented to illustrate the quality of the fit.

Table 1. Summary of the microstructure parameters measured on as-cast materials and after isothermal holding. The casting condition refers to the pouring temperature for standard experiments; "quench casting" is defined in the text.

As-cast microstructure			**Holding time (min.)**	**Evolved microstructure**	
Casting condition	**Structure**	**Grain size(μm)**		**Particle size (μm)**	**Morphology (roundness)**
quench-casting	Very coarse-grained, dendritic	1400	15	690	Solid network

725°C	Coarse-grained, dendritic	900	15	310	Solid network (2.7)
675°C	Medium-grained, dendritic	350	8	300	Rosette-like
			15	160	Irregular globular (2.4)
			30	140	Spherical globular (1.7)
650°C	Fine-grained, dendritic	200	3	202	Rosette-like (3.4)
			8	170	Irregular globular (2.1)
			15	102	Spherical globular (1.4)
			30	110	Spherical globular (1.3)
625°C	Fine rosette-like	180	8	130	Spherical globular (1.8)
			15	100	Spherical globular (1.4)
			30	105	Spherical globular (1.3)
650°C + G.R.	Fine globular	160	15	95	Spherical globular (1.3)